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(54) A spinning process for producing high strength, high modulus, low shrinkage synthetic yarns.

(57) A process for spinning an organic synthetic melt spinnable polymer is disclosed herein. The process includes the steps of: extruding the polymer through a spinneret; passing the filaments from the spinneret through an elongated zone; maintaining the filaments at a temperature above the glass transition temperature of the polymer within the zone; and thereafter converging the filaments. Alternatively, the process includes the steps of: extruding the polymer through a spinneret; providing an elongated zone having a length of at least 5 meters or means for controlling the temperature within said zone from a predetermined maximum to a predetermined minimum; passing the filaments through the zone; and thereafter converging the filaments.

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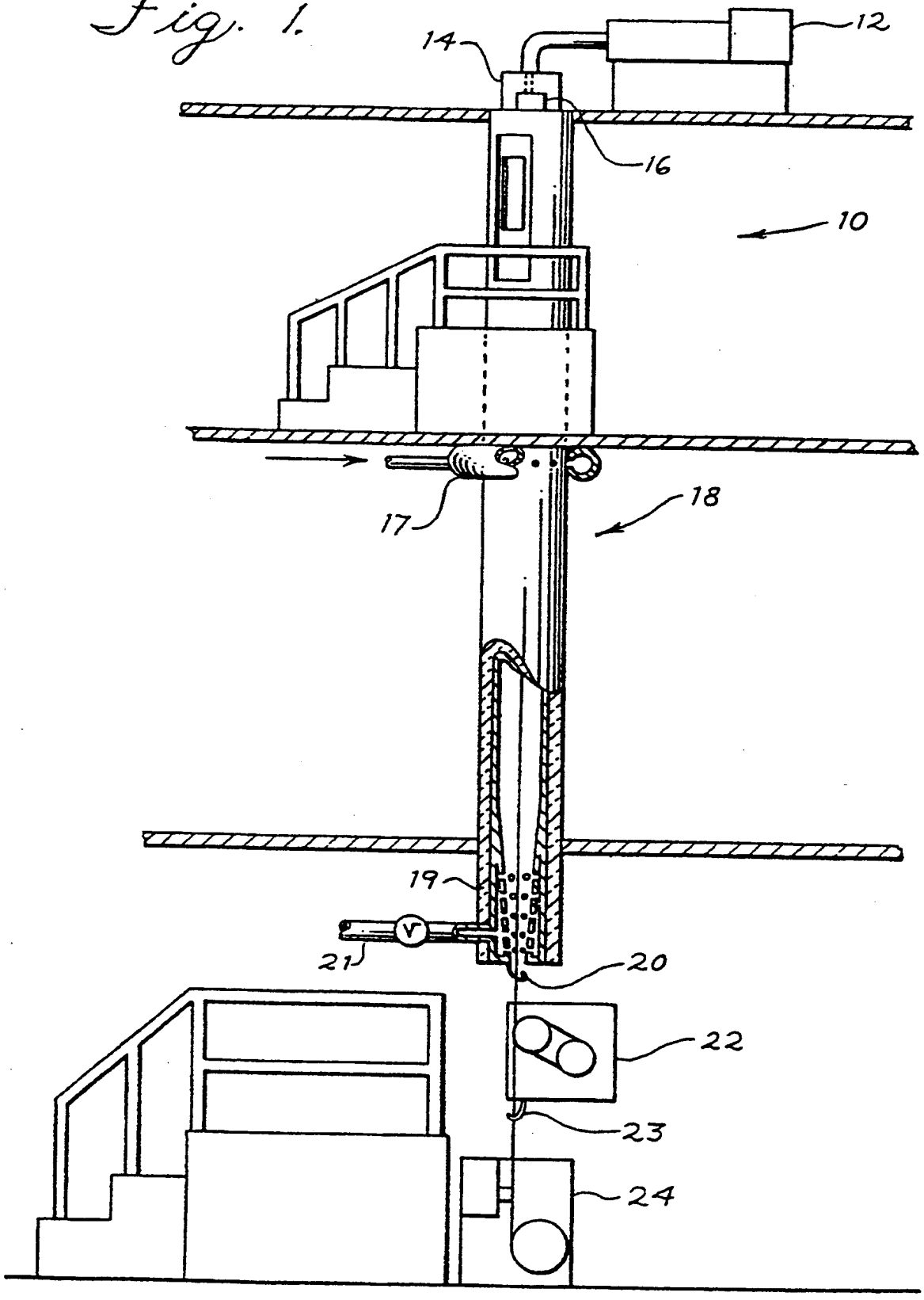
Fig. 1.



Field of the Invention

The instant invention is directed to a spinning process for producing high strength, high modulus, low shrinkage synthetic yarns.

Background of the Invention

Since fiber-forming, melt-spinnable, synthetic polymers were introduced, fiber manufacturers have looked for ways to increase the strength and stability properties of the fibers made from those polymers. The additional strength and stability properties of the fibers are needed so that applications beyond textile uses could be opened for their products. Such non-textile uses (also known as "industrial uses") include: tire cord; sewing thread; sail cloth; cloth, webs or mats used for road bed construction or other geo-textile applications; industrial belts; composite materials; architectural fabrics; reinforcement in hoses; laminated fabrics; ropes; and the like.

Originally, rayon was used in some of these industrial uses. Thereafter, nylon supplanted rayon as the material of choice. In the 1970's, conventional polyesters, such as polyethylene terephthalate, were introduced into competition against nylon. In about 1985, higher performance polyesters, i.e. higher strength and greater stability, were introduced.

A brief review of some of the patent prior art, summarized below, indicates that three general areas have been investigated as possible ways of enhancing the strength and stability properties of these synthetic fibers. Those general areas include: processes directed to drawing; processes directed to the polymer; and processes directed to the spinning. Hereinafter, the term "drawing" shall refer to the heating and stretching performed on an as-spun yarn. The term "treatment to the polymer" shall refer to those things done to the polymer prior to spinning. The term "spinning" shall refer to processes for forming filaments from polymer, but excluding drawing.

The processes directed to drawing are as follows:

In U. S. Patent No. 3,090,997, multistage drawing of polyamides, for use as tire cords, is disclosed. The fibers (nylon) are melt-spun in a conventional fashion. Thereafter, spun fibers are drawn in a three-stage process (drawn, then heated, then drawn again) to obtain a drawn nylon having the following properties: tenacity ranging from 10.4 to 11.1 grams per denier (gpd); elongation ranging from 12.9 to 17.1%; and initial modulus of 48 to 71 gpd/100%.

In U. S. Patent No. 3,303,169, there is disclosed a single-stage drawing process for polyamides that yields high modulus, high tenacity, and low shrinkage polyamide yarns. The spun polyamide is drawn and heated to at least 115°C to obtain a yarn having: tenacity in the range of 5 to 8.7 gpd; elongation ranging from 16.2 to 30.3%; initial modulus of 28 to 59gpd/100%; and shrinkage ranging from 3.5 to 15%.

In U. S. Patent No. 3,966,867, a two-stage drawing process for polyethylene terephthalate having a relative viscosity of 1.5 to 1.7 is disclosed. In the first stage, the fibers are subjected to a temperature between 70 and 100°C and a draw ratio of 3.8 to 4.2. In the second stage, the fibers are subjected to a temperature between 210 and 250°C and a draw ratio, in the aggregate of the first draw ratio and second draw ratio, in the range of 5.6 to 6.1. The drawn yarn obtained has the following properties: tenacity, 7.5 and 9.5 gpd; elongation, approximately 2 to 5% at a load of 5 gpd; elongation at break, 9 to 15%; and shrinkage, 1 to 4%.

In U. S. Patent No. 4,003,974, polyethylene terephthalate spun yarn, having an HRV of 24 to 28, is heated to 75 to 250°C while being drawn, is then passed over a heated draw roll, and finally relaxed. The drawn yarn has the following properties: tenacity, 7.5 to 9 gpd; shrinkage, about 4%; elongation at break, 12 to 20%; and load bearing capacity of 3 to 5 gpd at 7% elongation.

Those processes directed to enhancing yarn properties by treatment to the polymer are as follows:

In U. S. Patent Nos. 4,690,866 and 4,867,963, the intrinsic viscosity (I.V.) of the polyethylene terephthalate is greater than 0.90. In U. S. Patent No. 4,690,868, the as-spun (undrawn) fiber properties are as follows: elongation at break, 52 to 193%; birefriengence, 0.0626 to 0.136; and degree of crystallinity, 19.3 to 16.8%. The drawn fiber properties are as follows: tenacity, 5.9 to 8.3 gpd; elongation, 10.1 to 24.4%; and dry shrinkage (at 210°C), 0.5 to 10.3%. In U. S. Patent No. 4,867,916, the drawn fiber properties are follows: tenacity, about 8.5 gpd; elongation at break, about 9.9%; and shrinkage (at 177°C), about 5.7%.

Those processes directed to spinning are as follows:

In U. S Patent No. 3,053,611, polyethylene terephthalate after leaving the spinneret is heated to 220°C in a spinning shaft two meters long. Thereafter, cold water is sprayed onto the fibers in a second shaft. The fibers are taken up at a speed of 1,600 meters per minute (mpm) and are subsequently drawn to obtain a tenacity of 3.5 gpd.

In U. S. Patent No. 3,291,880, a polyamide is spun from a spinneret and then cooled to about 15°C, then the fiber is sprayed with live steam. The as-spun fiber has a low orientation and a low birefriengence.

In U. S. Patent No. 3,361,859, a synthetic organic polymer is spun into a fiber. As the fibers exit the spin-

neret, they are subjected to "controlled retarded cooling". This cooling is conducted over the first seven inches from the spinneret. At the top (i.e. adjacent the spinneret), the temperature is 300°C and at the bottom (i.e. approximately 7 inches from the spinneret), the minimum temperature is 132°C. The as-spun yarn has a low birefriengence (11 to 35 x 10^{-3}) and drawn yarn properties are as follows: tenacity, 6.9 to 9.4 gpd; initial modulus, 107 to 140 gpd/100%; and elongation at break, 7.7 to 9.9%.

In U. S. Patent Nos. 3,936,253 and 3,969,462, there is disclosed the use of a heated shroud (ranging in length from one-half foot to two feet) with temperatures ranging from about 115 to 460°C. In the former, the temperature is greater at the top of the shroud than at the bottom. The drawn yarn properties of the former are as follows: tenacity, 9.25 gpd; elongation, about 13.5%; and shrinkage, about 9.5%. In the latter, the temperature is constant within the shroud and the drawn yarn properties are as follows: tenacity, 8 to 11 gpd; and elongation at break, 12.5 to 13.2%.

In U. S. Patent No. 3,946,100, fibers are spun from a spinneret and solidified at a temperature below 80°C. The solidified fibers are then reheated to a temperature between the polymer's glass transition temperature (Tg) and its melting temperature. This heated fiber is withdrawn from the heating zone at a rate of between 1,000 to 6,000 meters per minute. Spun yarn properties are as follows: tenacity, 3.7 to 4.0 gpd; initial modulus, 70 to 76 gpd/100%; and birefriengence, 0.1188 to 0.1240.

In U.S. Patent No. 4,491,657, polyester multifilament yarn is melt-spun at high speed and solidified. Solidification occurs in a zone comprising, in series, a heating zone and a cooling zone. The heating zone is a barrel shaped heater (temperature ranging from the polymer's melting temperature to 400°C) ranging in length from 0.2 to 1.0 meters. The cooling zone is cooled by air at 10° to 40°C. Drawn yarn made by this process has the following properties: initial modulus, 90 - 130 gpd; and shrinkage (at 150°C) less than 8.7%.

In U. S. Patent No. 4,702,871, fiber is spun into a chamber having a subatmospheric pressure. Spun yarn properties are as follows: strength, 3.7 to 4.4 gpd; birefriengence, 104.4 to 125.8 (x 10^{-3}); and dry heat contraction, 4.2 to 5.9% at 160°C for 15 minutes.

In U. S. Patent No. 4,869,958, the fiber is spun in the absence of heat and then taken up. At this point, the fiber has a low degree of crystallinity, but it is highly oriented. Thereafter, the fiber is heat treated. The drawn fiber properties are as follows: tenacity, 4.9 to 5.2 gpd; initial modulus, 92.5 to 96.6 gpd/100%; and elongation, 28.5 to 32.5%.

The foregoing review of patents indicates that while some of the fibers produced by these various processes have high strength or low shrinkage properties, none of the foregoing patents teach of a yarn or a process for producing such a drawn yarn having the combination of high tenacity, high initial modulus, and low shrinkage.

The patents which come closest to teaching such a drawn yarn are U. S. Patent Nos. 4,101,525 and 4,195,052, related patents that are assigned to the assignee of the instant invention. In these patents, the polyester filaments (the polymer having an intrinsic viscosity of 0.5 to 2.0 deciliters per gram) are melt spun from a spinneret. Molten filaments are passed through a solidification zone where they are uniformly quenched and transformed into solid fibers. The solid fibers are drawn from the solidification zone under a substantial stress (0.015 to 0.15 gpd). These as-spun solid fibers exhibit a relatively high birefriengence (about 9 to 70 x 10^{-3}). The as-spun fibers are then drawn and subsequently heat treated. The drawn filament properties are as follows: tenacity, 7.5 to 10 gpd; initial modulus, 110 to 150 gpd/100%; and shrinkage, less than 8.5% in air at 175°C.

Summary of the Invention

A process for spinning an organic synthetic melt spinnable polymer is disclosed herein. The process includes the steps of: extruding the polymer through a spinneret; passing the filaments from the spinneret through an elongated zone; maintaining the filaments at a temperature above the glass transition temperature of the polymer over a distance of about 3 meters or greater within the zone; and thereafter converging the filaments.

Alternatively, the process includes the steps of: extruding the polymer through a spinneret; providing an elongated zone having a length of at least 5 meters or means for controlling the temperature within said zone from a predetermined maximum to a predetermined minimum; passing the filaments through the zone; and thereafter converging the filaments.

Description of the Drawing

For the purpose of illustrating the invention, there is shown in the drawing a schematic of the process which is presently preferred; it being understood, however, that this invention is not limited to the precise arrangement and instrumentalities shown.

Figure 1 is a schematic elevational view of the spinning process.
Figure 2 is a schematic elevational view of the drawing process.

Detailed Description of the Invention

High tenacity, high initial modulus, and low shrinkage drawn yarns and the process by which such yarns are spun are discussed hereinafter. The term "yarn" or "filament" or "fiber" shall refer to any fiber made from a melt spinnable synthetic organic polymer. Such polymers may include, but are not limited to, polyesters and polyamides. The invention, however, has particular relevance to polyesters such as, for example, polyethylene terephthalate (PET), blends of PET and polybutylene terephthalate (PBT), and PET cross-linked with multifunctional monomers (e.g. pentaerithritol). Any of the foregoing polymers may include conventional additives. The yarn I.V. (for PET based polymer) may be between 0.60 and 0.87. The instant invention, however, is not dependent upon the intrinsic viscosity (I.V.) of the polymer.

Referring to Figure 1, a spinning apparatus 10 is illustrated. A conventional extruder 12 for melting polymer chip is in fluid communication with a conventional spinning beam 14. Within spinning beam 14, there is a conventional spinning pack 16. Pack 16 may be of an annular design and it filters the polymer by passing the polymer through a bed of finely divided particles, as is well known in the art. Included as part of the pack 16 is a conventional spinneret (not shown). Flow rates of polymers through the pack may range from about 10 to 55 pounds per hour. The upper limit of 55 pounds is defined only by the physical dimensions of the pack 16 and greater flow rates may be obtained by the use of larger packs. The spun denier per filament (dpf) ranges from 3 to 20; it being found that the optimum properties and mechanical qualities for the yarn appear between 5 and 13 dpf.

Optionally, the fiber, as it leaves the spinneret, may be quenched with a hot inert gas (e.g. air). See U. S. Patent No. 4,378,325 which is incorporated herein by reference. Typically, the gas is about 230°C and is provided at about six standard cubic feet per minute (scfm). If the air is too hot, i.e. over 260°C, the spun yarn properties are significantly deteriorated.

Immediately below and snugly (i.e. airtight) mounted to spinning beam 14 is an elongated column 18. The column comprises an insulated tube having a length of about 5 meters or greater. Column length will be discussed in greater detail below. The tube's internal diameter is sufficiently large (e.g. twelve inches) so that all filaments from the spinneret may pass the length of the tube without obstruction. The column is equipped with a plurality of conventional band heaters so that the temperature within the tube can be controlled along its length. Column temperatures will be discussed in greater detail below. The column is, preferably, subdivided into a number of discrete temperature zones for the purpose of better temperature control. A total of 4 to 7 zones have been used. Optionally, the column 18 may include an air sparger 17 that is used to control temperature in the column. Sparger 17 is designed to evenly distribute an inert gas around the circumference of the column.

Inside the bottom-most end of the column 18 is a perforated, truncated cone 19, i.e. a means for reducing air turbulence. The cone 19, which is preferably three feet in length and having a diameter co-extensive with the tube diameter at its uppermost end and a diameter of about one half that at the bottom end, is used to exhaust air, via a valved exhaust port 21, from the bottom-most end of the tube so that movement in the thread line, due to air turbulence, is substantially reduced or eliminated completely.

Below the bottom-most end of the column, the thread line is converged. This convergence may be accomplished by a finish applicator 20. This is the first contact the yarn encounters after leaving the spinneret.

The length of the column, non-convergence of the individual filaments, and the air temperature profile within the column are of particular importance to the instant invention. With regard to the temperature profile, it is chosen so that the fibers are maintained at a temperature above their Tg over a significant length of the column (e.g. at least 3 meters). This temperature could be maintained over the entire length of the column, but the wound filaments would be unstable. Therefore, for practical reasons, the temperature within the column is reduced to below the Tg, so that the filaments will undergo no further changes in crystal structure before being wound up. Preferably, the temperature profile is chosen to reflect the temperature profile that would be established within the tube if no external heat was applied. However, the "no external heat" situation is impractical because of numerous variables that influence the column temperature. So, the temperature profile is controlled, preferably in a linear fashion, to eliminate temperature as a variable in the process.

The air temperature within the column is controlled by the use of the band heaters. Preferably, the column is divided into a plurality of sections and the air temperature in each section is controlled to a predetermined value. Thus, the temperature within the column can be varied over the length of the column. The temperature within the column may range from as high as the polymer spinning temperature to at or below the glass transition (Tg) temperature of the polymer (Tg for polyester is about 80°C). The polymer spinning temperature occurs around the spinneret, i.e. as the molten polymer exits the spinneret. However, air temperatures within the col-

umn are preferably controlled from about 155°C to about 50°C. At wind-up speeds less than 14,000 feet per minute, the first section adjacent the spinneret is preferably controlled to a temperature of about 155°C and the section furthest from the spinneret is controlled to about 50°C.

However, a linear temperature profile is not the only temperature pattern that will yield the beneficial results disclosed herein. At take-up (or wind-up) speeds greater than 14,000 fpm (4,300 mpm), the temperature profile (when the column is divided into four discrete zones) may be as follows: (starting from the spinneret down) the first zone - about 105°C to about 110°C; the second zone - about 110°C to about 115°C; the third zone - about 125° to about 130°C; and the fourth zone - 115°C to about 120°C.

With regard to column length, a minimum column length of five meters (with column temperature over the polymer's Tg for at least 3 meters) with filament convergence thereafter appears to be necessary for the instant invention. Column lengths between five and nine meters are suitable for the invention. The upper limit of nine meters is a practical limit and may be increased, room permitting. To optimize the tenacity properties, a column length of about seven meters is preferred.

The fibers are converged after exiting the column 18. This convergence may be accomplished by use of a finish applicator.

Following the first application of the finish (i.e. at finish applicator 20), the yarn is taken around a pair of godet rolls 22. Thereafter, a second application of finish may be made (i.e. at finish applicator 23). The first finish application may be made to reduce static electricity built up on the fibers. But this finish is sometimes thrown off as the fibers pass over the godet rolls. Thus, the finish may be reapplied after the godet rolls.

The fibers are then passed onto a conventional tension control winder 24. The wind-up speed is typically greater than 3,000 mpm (9,800 fpm) with a maximum speed of 5,800 mpm (19,000 fpm). An optimum range exists of about 10,500 to 13,500 fpm (about 3,200-4,100 mpm). The most preferred range exists between about 3200 and 3800 mpm (10,500 and 12,500 fpm). At speeds below 9,800 fpm (3,000 mpm), the yarn uniformity properties deteriorate.

The as spun polyester yarn produced by the foregoing process may be generally characterized as having relatively small crystals and a relatively high orientation. It is believed that these qualities of the as spun yarn enable the attainment of the unique drawn yarn properties discussed below.

To quantify the general characterization of the as spun polyester yarn, the small crystals are defined in terms of crystal size (measured in Å) and orientation is defined in one of the following terms: optical birefringence; amorphous birefringence; or crystal birefringence. Additionally, the spun polyester yarn is characterized in term of crystal size and long period spacing (the distance between crystals). In broad terms, the as spun polyester yarn may be characterized as having a crystal size less than 55Å and either an optical birefringence greater than 0.090 or an amorphous birefringence greater than 0.060 or a long period spacing of less than 300Å. More preferred, the as spun polyester yarn may be characterized as having a crystal size ranging from about 20 to about 55Å and either an optical birefringence ranging from about 0.090 to about 0.140 or an amorphous birefringence ranging from about 0.060 to about 0.100 or a long period spacing ranging from about 100 to about 250Å. Most preferred, the as spun polyester yarn may be characterized as having a crystal size ranging from about 43 to about 54Å and either an optical birefringence ranging from about 0.100 to about 0.130 or an amorphous birefringence ranging from about 0.060 to about 0.085 or a long period spacing ranging from about 140 to about 200Å.

As will be apparent to those of ordinary skill in the art, the crystal size of the spun yarn is about 1/3 that of conventional yarns in the optimum wind-up speed range. The crystal size increases with speed, but it still remains low. The spun amorphous orientation is very high, about twice normal. This spun yarn has such a high orientation and low shrinkage, that it could be used without any drawing.

In addition, the spun polyester yarn has the following properties: a crystal content (i.e. crystallinity level as determined by density) of 10 to 43%; a spun tenacity of about 1.7 to 5.0 gpd; a spun modulus in the range of 10 to 140 gpd/100%; a hot air shrinkage of about 5 to 45%; and an elongation of 50-160%.

Thereafter, the spun yarn is drawn. Refer to Figure 2. Either a one or two stage drawing operation may be used. However, it has been determined that a second stage offers little-to-no additional benefit. It is possible that the spinning operation may be coupled directly to a drawing operation (i.e., spin/draw process).

The as-pun yarn may be fed from a creel 30 onto a feed roll 14 that may be heated from ambient temperatures up to about 150°C. Thereafter, the fiber is fed onto a draw roll 38 which may be heated from ambient temperatures to approximately 255°C. If heated rolls are not available, a hot plate 36, which may be heated from 180° - 245°, may be used. The hot plate 36 (having a six inch curved contact surface) is placed in the draw zone, i.e., between feed roll 34 and draw roll 38. The draw speed ranges from 75 to 300 meters per minute. The typical draw ratio is about 1.65 (for spun yarn made at about 3,800 meters per minute). The optimum feed roll temperature, giving the highest tensile strength, was found to be about 90°C. The optimum draw roll temperature is about 245°C. If the hot plate is used, the optimum temperature is between about 240° - 245°C. The

draw roll temperature gives some control over hot air shrinkage. In general, low shrinkages are desirable as they give rise to the best treated cord stability ratings. However, at least one end use, sail cloth, requires higher drawn yarn shrinkages and these can be controlled with lower draw roll temperatures.

Based on the foregoing, the drawn fiber properties may be controlled as follows: Tenacity may range from 4.0 to 10.8 grams per denier. The elongation may range from 7% to approximately 80%. The initial secant modulus may range from 60 to 170 gpd/100%. The hot air shrinkage (at 177°C) is 6% to 15%. The denier of the fiber bundle may range from 125 to 1100 (the latter number may be obtained by plying tows together) and the denier per filament ranges from 1.5 to 6 dpf. Such a yarn could be used as the fibrous reinforcement of a rubber tire.

Polyester (i.e., PET) drawn yarns, made according to the process described above, can obtain an initial secant modulus greater than 150 grams per denier/100. Moreover, those yarns may also have a shrinkage of less than 8%, or those yarns may have a tenacity of greater than 7.5 grams per denier.

Another preferred embodiment of the drawn polyester yarn may be characterized as follows: a tenacity of at least 8.5 grams per denier; an initial modulus of at least 150 grams per denier/100%, and a shrinkage of less than 6%. Another preferred embodiment of the drawn polyester yarn may be characterized as follows: a tenacity of at least 10 grams per denier; an initial modulus of at least 120 grams per denier/100%; and a shrinkage of less than 6%. Yet another preferred embodiment of the drawn polyester yarn may be characterized as follows: a tenacity ranging from about 9 to about 9.5 grams per denier; an initial modulus ranging from about 150 to about 158 grams per denier/100%; and a shrinkage less than 7.5%.

Any drawn yarn, made according to the above described process, may be utilized in the following end uses: tire cord, sewing thread; sail cloth; cloth, webs or mats used in road bed construction or other geo-textile applications; industrial belts; composite materials; architectural fabrics; reinforcement in hoses; laminated fabrics; ropes; etc.

The following critical tests, which are used in the foregoing discussion of the invention and the subsequent examples, were performed as follows:

Tenacity refers to the "breaking tenacity" as defined in ASTM D-2256-80.

Initial modulus (or "initial secant modulus") is defined per ASTM D-2256-80, Section 10.3, except that the line representing the initial straight line portions of the stress-strain curve is specified as a secant line passing through the 0.5% and 1.0% elongation points on the stress-strain curve.

All other tensile properties are as defined in ASTM D-2256-80.

Shrinkage (HAS) is defined as the linear shrinkage in a hot air environment maintained at 177±1°C per ASTM D-885-85.

Density, crystal size, long period spacing, birefringence, and amorphous birefringence are the same as set forth in U.S. Patent No. 4,134,882 which is incorporated herein by reference. Specifically, each of the foregoing may be found in U.S. Patent No. 4,134,882 at or about: density - column 8, line 60; crystal size - column 9, line 6; long period spacing - column 7, line 62; crystal birefringence - column 11, line 12; and amorphous birefringence - column 11, line 27.

Birefringence (optical birefringence or Δ n) is as set forth in U.S. Patent No. 4,101,525 at column 5, lines 4-46. U.S. Patent No. 4,101,525 is incorporated herein by reference. "Bi CV" is the coefficient of variation of optical birefringence between filaments calculated from 10 measured filaments.

Other tests referred to herein are performed by conventional methods.

Reference should now be made to the Examples which will more fully illustrate the instant invention.

Example I

In the following set of experimental runs, a conventional polyester polymer (PET, IV-0.63) was spun. The spinning speeds were increased from 12,500 fpm to 19,000 fpm. The column length was 6.4 meters and divided into four temperature control zones. The temperature was controlled by measuring the air temperature close to the wall at the center of each zone. The polymer was extruded at a rate of 22.9 pounds per hour through a spinning beam at 285°C and a 40 hole spinneret (hole size 0.009 inches by 0.013 inches). The fibers were not quenched. The spun fibers were not drawn, but they were heat set. The results are set forth in TABLE 1.

TABLE I

	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8B
Spin Speed, fpm	12,500	13,500	14,500	15,500	16,500	17,500	18,500	19,000
Col - Top, °C	110	108	105	104	105	105	106	105
Temp. 2nd, °C	105	104	104	107	109	110	106	110
3rd, °C	131	130	129	132	132	132	130	133
Bottom, °C	109	107	105	111	111	111	109	119
Denier	340	310	290	270	255	240	225	220
dpf	8.5	7.8	7.2	6.8	6.4	6.0	5.6	5.5
"True Stress" at Break gpd	6.51	6.41	6.55	6.65	7.23	6.98	6.86	7.14
Spun: Denier	340	316	289	270	254	240	228	222
Tenacity, gpd	3.93	3.89	4.10	4.18	4.55	4.52	4.57	4.71
Elong, %	65.7	64.8	59.8	59.2	59.0	54.5	50.0	51.6
$T\sqrt{E}$	31.8	31.3	31.7	32.3	34.9	33.4	32.3	33.8
I.M.,gpd/100%	54.0	56.4	52.1	59.2	65.4	60.1	66.6	76.2
HAS, %-350°F	6.0	6.5	7.0	7.5	7.2	7.5	7.0	7.2
Uster, %	.96	1.29	1.14	1.28	1.33	1.59	1.34	1.52
Finish, %	.098	.358	.119	.168	.263	.037	.160	.267
IV	.623	.630	.629	.631	.630	.629	.626	.627
% Cryst.	34.2	35.3	37.2	39.0	40.3	42.2	43.2	43.3
$\Delta n \times 10^{-3}$	108	106	115	112	118	124	127	130
BiCV %	3.2	4.3	6.5	5.8	4.7	6.7	6.9	8.4
Density,gms/cc	1.3728	1.3742	1.3766	1.3788	1.3804	1.3827	1.3840	1.3841
Yield Point Tenacity, gpd	1.18	1.26	1.38	1.48	1.57	1.67	1.75	1.80
Heat-Set: Denier	338	308	287	271	252	240	226	231
Tenacity, gpd	4.06	4.19	4.26	4.34	4.33	4.46	4.65	4.64
Elong, %	62.3	58.6	53.2	51.0	49.5	46.6	44.4	45.1
$T\sqrt{E}$	32.0	32.1	31.1	31.0	30.5	30.5	31.0	31.2
I.M.,gpd/100%	60.2	62.2	66.3	70.0	68.8	64.0	73.2	72.6
HAS, %-350°F	2.0	2.2	2.8	2.8	3.0	3.2	3.0	2.5
% Cryst.	55.7	55.9	56.6	56.9	56.9	57.0	57.3	57.2
$\Delta n \times 10^{-3}$	152	142	143	145	150	146	156	160
BiCV %	5.8	7.9	7.9	6.3	7.0	6.5	9.1	6.3
Density,gms/cc	1.3996	1.3999	1.4007	1.4011	1.4011	1.4013	1.4016	1.4015
Yield Point Tenacity, gpd	0.89	0.97	1.04	1.11	1.19	1.25	1.33	1.30

Example II

In the following set of experimental runs, a conventional polyester (PET, IV-0.63) was spun. The column temperatures were varied as indicated (air temperature, center of zones). The column length was 6.4 meters. The polymer was extruded at a rate of 23.1 pounds per hour through a spinning beam at 300°C and a 72 hole spinneret (hole size 0.009 inches by 0.012 inches). The fibers were not quenched. The spun fibers were subsequently drawn (as indicated). The results are set forth in TABLE II.

TABLE II

	No. 1	No. 4	No. 5	No. 2	No. 3	No. 6	No. 7
Spin Speed-fpm-1000's	10.5	10.5	10.5	12.5	12.5	12.5	12.5
Hot Quench-scfm/°C	6/230°						
Air Bleed*-scfm/°C	30/35°						
Col. Temp Top °C	70	68	120	80	98	121	135
2nd °C	83	101	99	81	88	101	107
3rd °C	75	88	85	75	78	86	88
Bottom °C	62	72	79	64	65	80	81
Spun: Denier	370	367	369	344	342	342	342
Tenacity-gpd	2.87	3.68	3.77	3.50	3.72	3.86	3.75
Elong-%	122	81.8	83.2	82.6	79.6	70.9	69.0
I.M.-gpd/100%	63	93	93	86	86	73	75
HAS-% 350°F	65.5	27.2	41.0	49.5	42.0	11.2	9.5
Uster-%	1.38	1.14	1.41	.99	1.13	1.23	2.29
Finish-%	1.82	.44	.74	.96	.85	.50	.54
IV	.63	.64	.64	.64	.64	.64	.64
$\Delta n \times 10^{-3}$	78	115	113	105	111	107	106
% Cryst.	11.0	17.9	16.6	14.8	15.9	20.5	24.7
Max Draw Ratio (D.R.)	1.70	1.80	1.80	1.60	1.57	1.77	1.74
Denier	224	210	213	218	227	202	206
Tenacity-gpd	5.60	8.72	8.63	7.31	7.04	8.74	8.67
Elong-%	18.4	8.9	8.6	11.0	11.6	7.5	8.1
I.M.-gpd/100%	92	137	133	127	110	146	140
HAS-% 350°F	6.2	10.0	9.8	9.2	7.8	10.0	10.0
Max D.R. - .03	1.65	1.77	1.77	1.54	1.54	1.74	1.72
Denier	230	214	217	227	231	205	205
Tenacity-gpd	5.34	8.30	8.72	7.04	7.09	8.61	8.31
Elong-%	19.9	9.3	9.2	13.1	13.1	7.7	7.6
I.M.-gpd/100%	82	120	137	123	107	145	124
HAS-% 350°F	6.0	9.8	10.0	9.0	7.8	10.2	10.0

*Air sparger, item 17, Figure 1

In the above set of experimental runs (i.e., those set forth in TABLE II), Nos. 4, 5, 6 and 7 represent the instant invention.

Example III

In the following sets of experimental runs, conventional polyester (PET, IV-0.63) was spun. The fibers were wound up at a rate of 10,500 fpm. The polymer was extruded at a rate of 19.5 pounds per hour through a 72 hole spinneret (hole size 0.009 inches by 0.012 inches) and a spinning beam at 300°C. The fibers were quenched with 6.5 scfm air at 232°C. The column was 6.4 meters long and divided into 4 sections having the following air temperature profile (in descending order): 135°C; 111°C; 92°C; and 83°C at the center of the zones. The spun yarn had the following properties: denier - 334; tenacity - 4.09 gpd; elongation 71.7%; initial modulus - 55.0 gpd/100%; hot air shrinkage - 11.8% at 350°F.; Uster 1.10; I.V. -0.647; FOY - 0.35%; birefringence - 110 x 10^{-3}; and crystallinity - 21.6%.

In TABLE IIIA, the effect of draw ratio on drawn yarn properties is illustrated.

TABLE IIIA

Draw Ratio	1.65	1.60	1.54
Denier	209	218	226
Tenacity gpd	8.15	7.53	7.12
Elongation %	8.4	8.9	10.4
Initial Modulus gpd/100%	123	115	115
Hot Air Shrinkage % 350°F	12.0	12.4	12.0

In Table IIIB, the effect of the heating method during stretching is illustrated (the draw ratio was 1.65 and the yarn was not relaxed).

TABLE IIIB

Denier	Tenacity	Elongation	Initial Modulus	Hot Air Shrinkage 350°F	Feed Roll Temp.	Hot Plate Temp.	Draw Roll Temp.
	gpd	%	gpd/100%	%	°C	°C	°C
334	4.09	71.7	55	11.8		(As Spun)	
209	8.15	8.4	123	12.0	Amb	245	Amb
214	6.67	9.2	95	19.0	78	Amb	Amb
212	8.05	9.3	86	8.0	78	245	Amb
209	8.05	9.0	93	9.0	78	Amb	200
211	8.45	9.1	110	9.2	78	245	200
211	7.96	8.8	110	9.2	100	245	200
211	8.18	9.2	108	9.2	120	245	200

In Table IIIC, the effect of higher drawing temperatures and draw ratios is illustrated (the feed roll is at ambient temperature and the draw roll is at 240°C).

TABLE IIIC

Draw Ratio		1.76	1.72	1.70	1.67	1.64	1.61
Denier		195	194	199	203	209	208
Tenacity	gpd	9.50	9.22	8.89	8.73	7.76	6.71
Elorgation	%	6.1	6.1	6.3	6.7	6.6	7.5
Hot Air Shrinkage	%-350°F	6.8	7.0	6.8	6.5	6.8	6.5

Example IV

In the following set of experimental runs, a conventional polyester (PET, IV-0.92) was spun. In runs Nos. 1-5, the fibers were spun and drawn in accordance with the methods set forth in U. S. Patent Nos. 4,101,525 and 4,195,052. Nos. 6-9 were made as follows:

PET with a molecular weight characterized by an I.V. of 0.92 was dried to a moisture level of 0.001% or less. This polymer was melted and heated to a temperature of 295°C in an extruder and subsequently forwarded to a spinning pack by a metering pump. This pack was of an annular design, and provided filtration of the polymer by passing it through a bed of finely divided metal particles. After filtration the polymer was extruded through an 80 hole spinneret. Each spinneret hole had a round cross section with a diameter of 0.457 mm and a capillary length of 0.610 mm.

An insulated heated tube 9 meters in length was mounted snugly below the pack and the multifilament spinning threadline passed through the entire length of this tube before being converged or coming into contact with any guide surfaces. The tube was divided down its length into seven zones for the purposes of temperature control. Individual controllers were used to set the air temperature at the center of each of these zones. Using a combination of process heat and the external heaters around the tube, individual controller settings were selected to arrive at a uniform air temperature profile down the vertical distance of this tube. In a typical situation

the air temperature was 155°C at the top zone of the tube and the temperature was reduced in an approximately uniform gradient to 50°C at the bottom.

Approximately 10 cm below the tube the threadline was brought into contact with a finish applicator which also served as the convergence guide and the first contact that the yarn encountered. At the exit of the tube the cross section of the un-converged yarn was very small due to the proximity of the finish guide. This permitted a very small aperture to be used, thus minimizing the amount of hot air lost from the tube.

Following the application of spin finish the yarn was taken to a pair of godet rolls and then to a tension controlled winder. Wind up speeds were typically in the range 3200 - 4100 mpm.

Drawing of this yarn was effected in a second step, in which the as spun yarn was passed over one set of pretension rolls to a heated feed roll maintained at a temperature set between 80 and 150°C. The yarn was then drawn between these rolls and a set of draw rolls maintained at a set point chosen in the range 180 to 255°C. A typical draw ratio for a spun yarn made at 3800 mpm would be 1.65, with samples spun at higher and lower speeds requiring lower or higher draw ratios, respectively.

The results are set forth in TABLE IV.

TABLE IV

			Feed Roll Temperature °C					
			25			90		
No.	Spinning Speed (fpm)	Spun Yarn Birefringence x10-3	Tenacity gpd	Initial Modulus gpd/100%	Drawn Yarn Shrinkage % 350°F	Tenacity gpd	Initial Modulus gpd/100%	Drawn Yarn Shrinkage % 350°F
1	5000	21.9	7.94	115.00	7.30	5.96	78.00	5.30
2	6000	30.1	7.85	118.00	7.00	6.90	103.00	6.70
3	7000	45.2	8.36	120.00	7.00	7.21	108.00	6.50
4	8000	60.5	8.51	130.00	7.80	7.31	113.00	6.00
5	9000	78	8.56	122.00	6.80	7.67	110.00	6.00
6	10500	104	9.52	158.00	7.50	10.94	173.00	7.30
7	11500	115	9.03	150.00	6.80	9.52	152.00	7.00
8	12500	121	9.08	152.00	7.50	9.53	160.00	7.30
9	13500	119	9.32	154.00	6.00	9.58	161.00	6.70

EXAMPLE V

Polyester with a molecular weight characterized by an I.V. of 0.92 was dried to a moisture level of 0.001%. This polymer was melted and heated to a temperature of 295°C in an extruder and the melt subsequently forwarded to a spinning pack by a metering pump. After filtration in a bed of finely divided metal particles, the polymer was extruded through an 80 hole spinneret. Each spinneret hole had a diameter of 0.457.mm and a capillary length of 0.610 mm. On extrusion the measured I.V. of this polymer was 0.84.

The extruded polymer was spun into heated cylindrical cavity 9 meters in length. An approximately linear temperature profile (gradient) was maintained over the length of this tube. At the center of the top zone the air temperature was 155°C and at the bottom of the tube this temperature was 50°C. The multifilament yarn bundle was not converged until it came in contact with a finish guide just below the exit of the heated tube. From this point the yarn was advanced by a pair of godet rolls to a tension controlled winder. Under these conditions a series of four spun yarns were made at different spinning (wind-up) speeds. These yarns are referred to as examples A through D in Table V. A.

In another series of experiments the heated tube was shortened by taking out some of its removable sections. Examples E and F in Table V. A were spun through 7 and 5 meter columns. Other polymers with different molecular weights (I.V.'s) were also spun on this system to give Examples G and H. Example I in Table VA illustrates a case in which lower column temperatures were used. In this case a linear gradient from 125°C to 50°C was established down the column.

All spun yarns in the series A through I were drawn in a single stage process using an ambient feed roll and a 245°C draw roll.

In a further series of tests the same spun yarn which was described in Example A was drawn using different feed roll temperatures. The results from testing these yarns are given in Examples A, J and K in Table V. B.

TABLE V. A

Example	Spinning Conds: Length	Spin Speed mpm	Temp °C	Spun IV	Spun Yarn: Birr	Spun Yarn: Cryst %	Draw Ratio	Drawn Yarn: Ten gpd	Drawn Yarn: I.M. gpd/100%	Drawn Yarn: HAS %-350°F
A	9	3200	155	0.84	.104	30.5	1.89	9.52	158	7.5
B	9	3500	155	0.84	.115	34.4	1.79	9.03	150	6.8
C	9	3800	155	0.84	.121	35.9	1.74	9.08	152	7.5
D	9	4100	155	0.84	.119	38.9	1.72	9.32	154	6.0
E	7	3200	155	0.84	.101	30.1	1.79	8.99	142	7.3
F	5	3200	155	0.84	.073	25.0	1.98	9.52	159	7.0
G	9	3200	155	0.76	.110	34.0	1.65	8.63	123	6.0
H	9	3200	155	0.66	.102	22.9	1.57	7.25	110	5.0
I	9	4100	125	0.84	.120	31.9	1.53	7.34	116	5.3

TABLE V. B

Example	Feed Roll Temp °C	Draw Ratio	Drawn Tenacity gpd	Drawn I Modulus gpd/100%	Hot Air Shrink %-350°F
A	25	1.89	9.52	158	7.5
J	90	1.82	10.94	173	7.7
K	150	1.87	10.30	158	7.4

EXAMPLE VI

In the following experimental run, a conventional polymer, nylon, was spun according to the inventive process and compared to nylon made by conventional processes.

The nylon made by the inventive process was spun under the following conditions: troughput- 37 lbs. per hour; spinning speed - 2,362 fpm; denier - 3500; number of filaments - 68; spun relative viscosity - 3.21 (H_2SO_4) or 68.4 (HC00H equiv.) quench air - 72 scfm; winding tension 80g; column length - 24 ft; column temperature top 240°C and bottom 48°C. The as-spun properties of this yarn were as follows: tenacity - 0.95 gpd; elongation 235%; $TE^{1/2}$ - 14.6. Thereafter the yarn was drawn under the following conditions: draw ratio 3.03; draw temperature 90°C. The drawn yarn properties are as follows: tenacity 6.2 gpd; elongation -70%; $TE^{1/2}$ - 52; 10% modulus - 0.87 gpd; hot air shrinkage (HAS) at 400°F - 1.4%.

One comparative nylon was spun in the following conventional fashion: throughput - 23.4 lbs. per hour; spinning speed - 843 fpm; denier - 5556; number of filaments - 180; spun relative viscosity - 3.3 (H_2SO_4) or 72.1 (HCOOH equiv.); quench - 150 scfm. Thereafter, the yarn was drawn under the following conditions: Draw ratio - 2.01; draw temperature - 90°C. The drawn yarn properties are as follows: tenacity 3.8 gpd; elongation - 89%; $TE^{1/2}$ - 33; 10% modulus - .55 gpd.

Another comparative yarn was spun in the following conventional fashion: throughput - 57.5 lbs. per hour; spinning speed - 1048 fpm; denier - 12400; number of filaments - 240; spun relative viscosity - 42 (HC00H equiv.); quench air - 150 scfm. Thereafter, the yarn was drawn under the following conditions: draw ratio - 3.60; draw temperature - 110°C. The drawn yarn properties are as follows: tenacity - 3.6 gpd; elongation - 70%; $TE^{1/2}$ - 30.1; modulus at 10% elongation - 0.8 gpd; HAS (at 400°F) - 2.0%.

EXAMPLE VII

In the following experimental runs, low I.V. (e.g. 0.63) and high I.V. (e.g. 0.92) conventional polyester (i.e. PET) as spun yarn is compared with as spun yarn set forth in U.S. Patent No. 4,134,882. Examples 1-8 are

low I.V. polyester (PET) and are made in the manner set forth in Example I. Examples 9-11 are high I.V. polyester (PET) and are made in the manner set forth in Example V. Examples 12-17 correspond to Examples 1, 5, 12, 17, 36 and 20 of U.S. Patent No. 4,134,882.

For each example, the spinning speed (fpm), density (gms/cc), crystal size (Å, 010), long period spacing (LPS), birefringence (biref.), crystal birefringence and amorphous birefringence are given. The results are set forth in Table VII.

TABLE VII

No.	Spin Speed (fpm)	Density gms/cc	CS 010 Å	LPS Å	Biref.	Crystal Biref.	Amorphous Biref.
1	12500	1.3728	45	147	0.1080	0.1982	0.067
2	13500	1.3742	45	160	0.1060	0.1994	0.061
3	14500	1.3766	47	155	0.1150	0.2004	0.070
4	15500	1.3788	50	158	0.1120	0.2021	0.060
5	16500	1.3804	51	145	0.1180	0.2035	0.066
6	17500	1.3827	53	152	0.1240	0.2042	0.071
7	18500	1.3840	55	147	0.1270	0.2055	0.073
8	19000	1.3841	54	150	0.1300	0.2052	0.078
9	10000	1.3485	21	192	0.0761	0.1824	0.063
10	10000	1.3653	43	192	0.1047	0.1930	0.075
11	12500	1.3749	52	183	0.1215	0.1994	0.083
12	16500	1.3700	61	313	0.0958	0.2010	0.045
13	18000	1.3770	73	329	0.1082	0.2010	0.057
14	19500	1.3887	72	325	0.1153	0.2030	0.054
15	21000	1.3868	68	330	0.1241	0.2050	0.063
16	21000	1.3835	64		0.1236	0.1980	0.073
17	16500	1.3766	65		0.0965	0.2060	0.038

The present invention may be embodied in other specific forms without departing from the spirit or essential attributes thereof and, accordingly, reference should be made to the appended claims, rather than to the foregoing specification, as indicating the scope of the invention.

Claims

1. A process for spinning an organic synthetic melt spinnable polymer comprising the steps of:
   extruding the polymer through a spinneret;
   passing the filaments from the spinneret through an elongated zone;
   maintaining the filaments at a temperature above the glass transition of the polymer over a distance of about 3 meters or greater within the zone; and
   thereafter converging the filaments.

2. The process according to claim 1 further comprising the step of: winding up the filaments after converging.

3. The process according to claim 1 further comprising the step of: spinning the filaments from the spinneret so that the filaments have a spun denier per filament of 3-20.

4. The process according to claim 1 further comprising the step of: quenching the filaments with a hot gas as the filaments leave the spinneret.

5. The process according to claim 4 further comprising the step of: quenching the filaments with a hot gas having temperature no greater than 260°C.

6. The process according to claim 5 further comprising the step of: quenching the filaments with a hot gas having a temperature of 230°C.

7. The process according to claim 1 further comprising the step of: passing the filaments from the spinneret through the elongated zone, said zone having a length of at least 5 meters wherein temperatures in the zone are controlled over the length of the zone from a maximum of the polymer spinning temperature to a minimum of ambient temperature.

8. The process according to claim 7 further comprising the steps of: passing the filaments from the spinneret through the elongated zone wherein temperatures in the zone are controlled from about 155°C proximal the spinneret to about 50°C distal the spinneret.

9. The process according to claim 8 further comprising the step of: passing the filaments from the spinneret through the elongated zone wherein temperatures in the zone are controlled from about 155°C proximal the spinneret to about 50°C distal the spinneret and the temperature between said proximal and distal points decreases in a generally linear fashion.

10. The process according to claim 1 further comprising the step of: passing the filaments from the spinneret through the elongated zone having a length ranging from about 5 to about 9 meters.

11. The process according to claim 2 further comprising the step of: winding up the filaments at a rate of at least 5,000 feet per minute.

12. The process according to claim 2 further comprising the step of: winding up the filaments at a rate of about 5000 to about 19000 feet per minute.

13. The process according to claim 2 further comprising the step of: winding up the filament at a rate of about 10,500 to about 13,500 feet per minute.

14. The process according to claim 2 further comprising the step of: winding up the filament at a rate of greater than 14,000 feet per minute after passing the filaments from the spinneret through said elongated zone having been divided into four portions wherein the temperature in the first portion adjacent the spinneret has a temperature ranging from about 105°C to about 110°C; the temperature in the second portion adjacent the first zone has a temperature ranging from about 110°C to about 115°C; the temperature in the third portion adjacent the second zone has a temperature ranging from about 125°C to about 130°C; and the temperature in the fourth portion adjacent the third zone has a temperature ranging from about 115°C to about 120°C.

15. A process for spinning an organic synthetic melt spinnable polymer comprising the steps of:
extruding the polymer through a filament forming means;
providing an elongated zone having a length of at least 5 meters;
passing the filaments from said filament forming means through said elongated zone; and
thereafter converging the filaments.

16. A process for spinning an organic synthetic melt spinnable polymer comprising the steps of:
extruding the polymer through a filament forming means;
providing an elongated zone having means for controlling the temperature within said zone from a predetermined maximum to a predetermined minimum;
passing the filaments from said filament forming means through said elongated zone; and
thereafter converging the filaments.

Fig. 1.

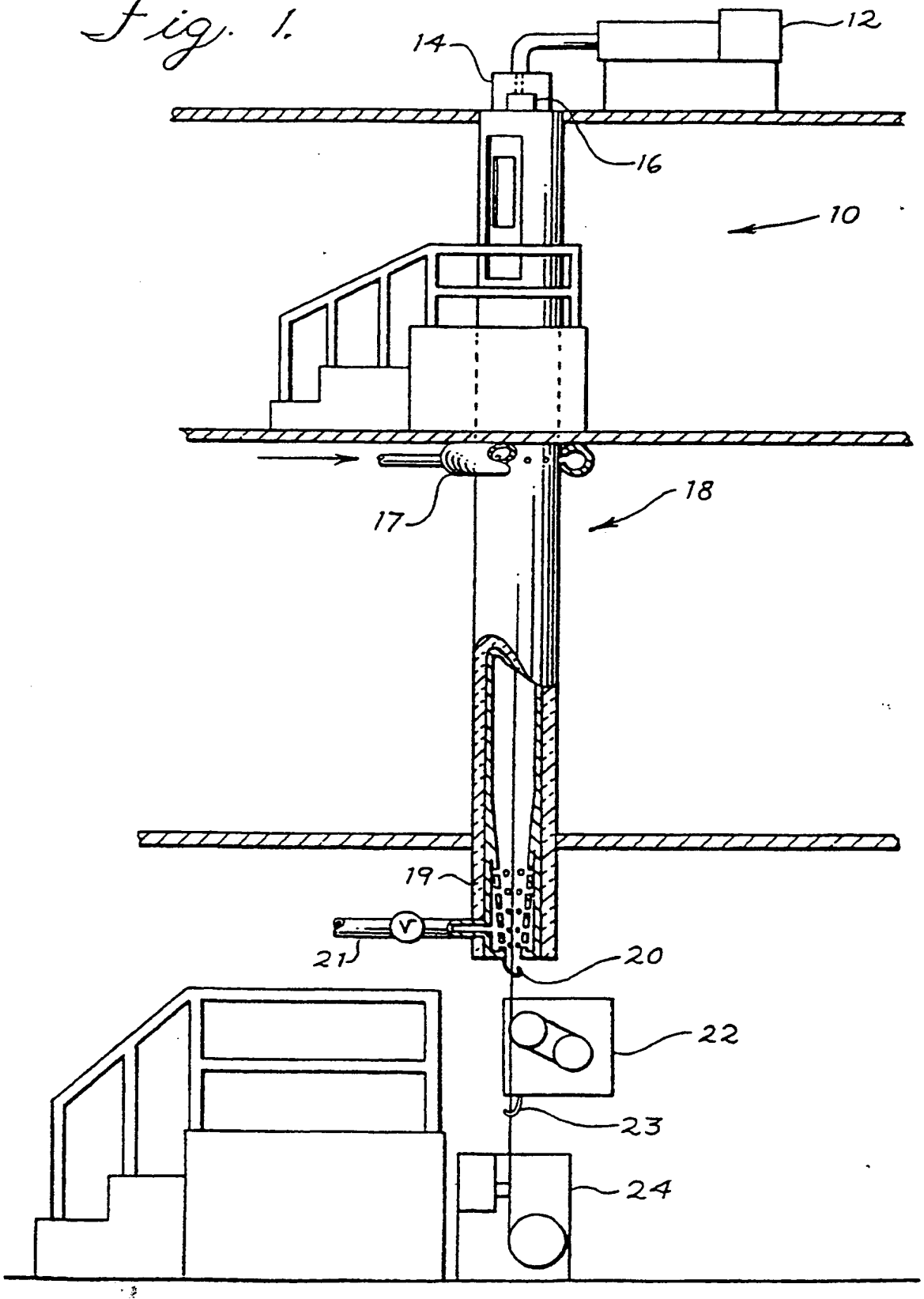
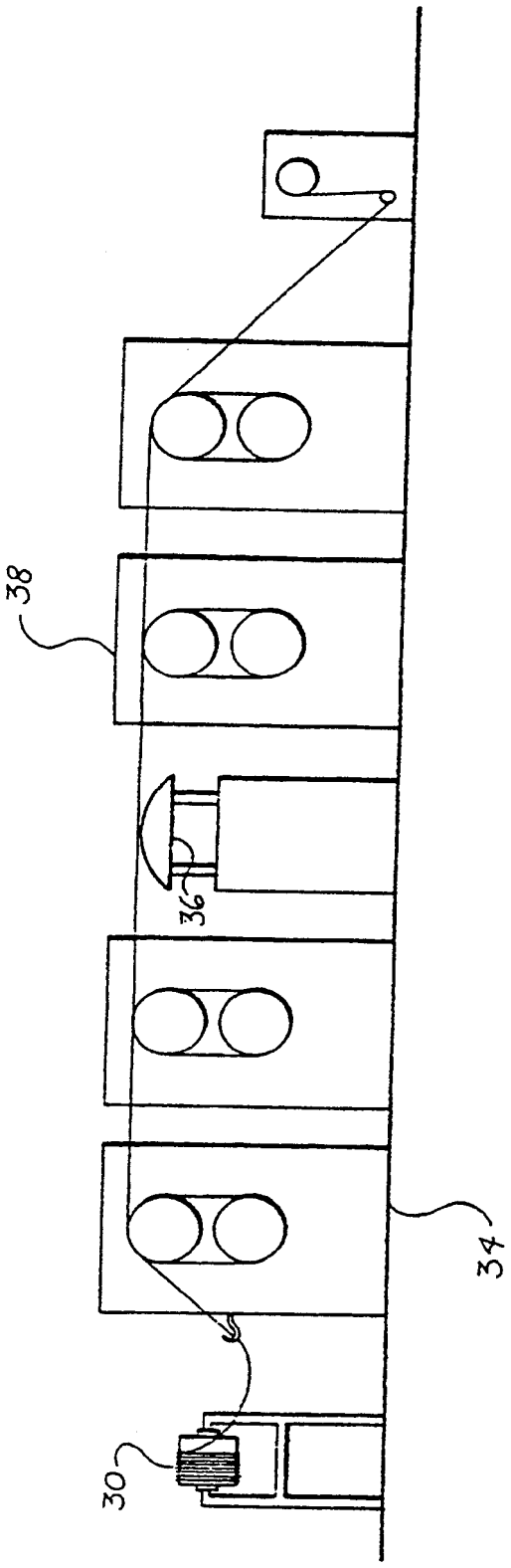


Fig. 2.



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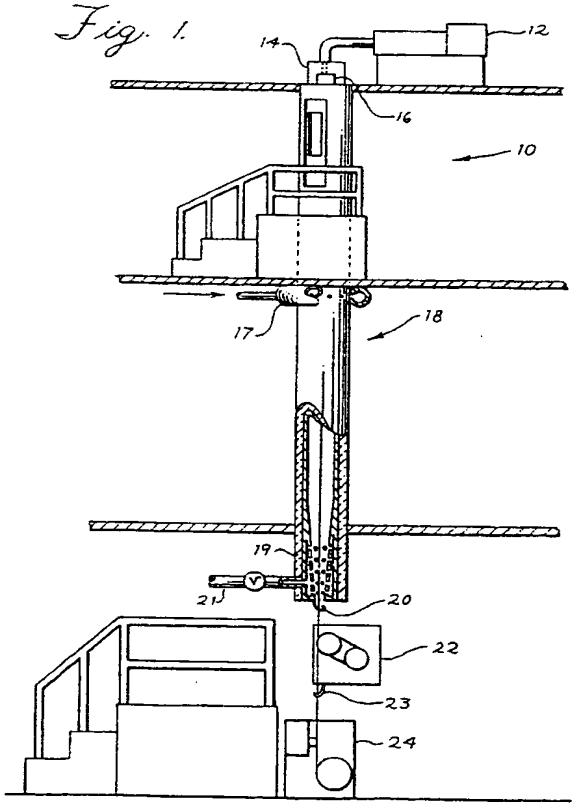
(54) A spinning process for producing high strength, high modulus, low shrinkage synthetic yarns.

(57) A process for spinning an organic synthetic melt spinnable polymer is disclosed herein. The process includes the steps of: extruding the polymer through a spinneret (16); passing the filaments from the spinneret through an elongated zone (18); maintaining the filaments at a temperature above the glass transition temperature of the polymer within the zone; and thereafter converging the filaments. Alternatively, the process includes the steps of: extruding the polymer through a spinneret; providing an elongated zone having a length of at least 5 meters or means for controlling the temperature within said zone from a predetermined maximum to a predetermined minimum; passing the filaments through the zone; and thereafter converging the filaments.

Fig. 1.



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EUROPEAN SEARCH REPORT

Application Number

EP 91 30 4190

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	US-A-3 053 611 (WOLFGANG GRIEHL) * column 1, line 61 - column 2, line 52; claims; examples *	1-7, 10-12, 15,16	D01D5/092 D01F6/62 D01F6/60
A	EP-A-0 341 920 (TORAY INDUSTRIES INC.) * claim 5 *	1-16	
D,A	US-A-4 491 657 (ISOO SAITO) * claim 2 *	1-16	
A	EP-A-0 034 880 (IMPERIAL CHEMICAL INDUSTRIES LTD) * claim 1 *	1-16	
A	PATENT ABSTRACTS OF JAPAN vol. 7, no. 139 (C-171)(1284) 17 June 1983 & JP-A-58 054 020 (TEIJIN KK) 30 March 1983 * abstract *	1-16	
A	WORLD PATENTS INDEX Week 8013, Derwent Publications Ltd., London, GB; AN 80-22821C & JP-A-55 022 012 (TORAY IND INC) 16 February 1980 * abstract *	1-16	TECHNICAL FIELDS SEARCHED (Int. Cl.5) D01D D01F
A	WORLD PATENTS INDEX Week 7950, Derwent Publications Ltd., London, GB; AN 79-90510B & JP-B-54 038 213 (TEIJIN KK) 20 November 1979 * abstract *	1-16	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	27 JANUARY 1992	TARRIDA TORRELL J.B.

CATEGORY OF CITED DOCUMENTS

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